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HIGH STRENGTH BOLT HAVING EXCELLENT DELAYED FRACTURE RESISTANCE
[TAIOKUREHAKAISEI NI SUGURETA KOUKYODO BORUTO]

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[Scope of Patent Claims]

[Claim 1] A high strength bolt having excellent delayed fracture resistance, characterized in that a high strength wire material which is constituted of a steel containing C: 0.5 to 1.0 % (% by mass, the same applies below), restrains the generation of one, two, or more structures selected from the group consisting of pro-eutectoid ferrite, pro-eutectoid cementite, bainite, and martensite in order to make the area rate of pearlite structure having a pearlite lamelar spacing of 200 nm or less to be 80 % or more and is made to have a tensile strength of 1,200 N/mm^2 or more and excellent delayed fracture resistance by using strong wire-drawing, the high strength wire material is cut into a predetermined length, and ends are threaded by thread rolling or cutting.

[Claim 2] The high strength bolt according to Claim 1, characterized in that a high strength wire material containing Si: 2.0 % or less (but not 0 %) and/or Co: 0.5 % or less (but not 0 %) is used.

[Claim 3] The high strength bolt according to either Claim 1 or 2, characterized in that a high strength wire material containing Cr: 1.0 % or less (but not 0 %) is used.

[Claim 4] A high strength bolt having excellent delayed fracture resistance, characterized in that a high strength wire material

which is constituted of a steel containing C: 0.5 to 1.0 % (% by mass, the same applies below), restrains the generation of one, two, or more structures selected from the group consisting of pro-eutectoid ferrite, pro-eutectoid cementite, bainite, and martensite in order to make the area rate of pearlite structure having a pearlite lamelar spacing of 200 nm or less to be 80 % or more and is made to have a tensile strength of 1,200 N/mm^2 or more and excellent delayed fracture resistance by using strong wire-drawing, the high strength wire material is cut into a predetermined length, a bolt head is formed on one end thereof by warm forging, and the other end is thread-rolled or threaded.

[Claim 5] The high strength bolt according to Claim 4, characterized in that a high strength wire material containing Si: 2.0 % or less (but not 0 %) and/or Co: 0.5 % or less (but not 0 %) is used.

[Claim 6] The high strength bolt according to either Claim 4 or 5, characterized in that a high strength wire material containing Cr: 1.0 % or less (but not 0 %) is used.

[Detailed Description of the Invention]

[0001]

[Technical Field of the Invention] The present invention relates to a high strength bolt which is used for an automobile or various other industrial machineries and, in particular, to a

high strength bolt having excellent delayed fracture resistance while having a strength (tensile strength) of 1,200 N/mm^2 or more.

[0002]

[Prior Art] Medium carbon alloy steels (such as SCM 435, SCM 440 and SCr 440) are known as commonly-used steels for the production of a high strength bolt and ensure a required strength by the quench hardening and tempering thereof. However, in the case where a conventional high strength bolt, which is used for the production of an automobile or various other industrial machineries, has a tensile strength of over 1,200 N/mm^2, it is likely to cause a delayed fracture and the applicable conditions are therefore limited.

[0003] The delayed fracture includes both phenomena occurring in corrosive and non-corrosive environments, and the causes of its occurrence are said to be complicated due to various factors and it is therefore difficult to precisely specify the causes. The regulators which are possibly responsible for the delayed fracture include tempering temperature, material structure, material hardness, grain size, various alloy elements, and others, but there is currently no effective means to prevent the delayed fracture, and various methods are studied by trial and error even now.

[0004] In order to improve the delayed fracture resistance, many methods have been proposed, such as Japanese Unexamined Patent

Publication No. Shōwa 60-114551, Japanese Unexamined Patent Publication No. Heisei 2-267243 and Japanese Unexamined Patent Publication No. Heisei 3-243745. These methods intend to develop a steel for the production of a high strength bolt, wherein a desired delayed fracture resistance is ensured even at a tensile strength of 1,400 MPa or more by adjusting the contents of various major alloy elements. However, even the above described methods unable the risk of occurrence of the delayed fracture to be completely eliminated and the application thereof still remains in extremely limited areas.

[0005]

[Problems to be Solved by the Invention] In view of the above described situation, the present invention intends to provide a high strength bolt having excellent delayed fracture resistance while having a tensile strength of 1,200 N/mm^2 or more.

[0006]

[Means of Solving the Problems] The inventive high strength bolt which attains the above described object is characterized in that a high strength wire material which is constituted of a steel containing C: 0.5 to 1.0 % (% by mass, the same applies below), restrains the generation of one or more structures selected from the group consisting of pro-eutectoid ferrite, pro-eutectoid cementite, bainite and martensite in order to make the area rate of pearlite structure having a pearlite lamelar

spacing of 200 nm or less to be 80 % or more and is made to have a tensile strength of 1,200 N/mm^2 or more and excellent delayed fracture resistance by using strong wire-drawing, the high strength wire material is cut into a predetermined length and ends are threaded by thread rolling or cutting.

[0007] The above described object of the present invention can also be attained by a high strength bolt which is characterized in that a high strength wire material which is constituted of a steel containing C: 0.5 to 1.0 % (% by mass, the same applies below), restrains the generation of one or more structures selected from the group consisting of pro-eutectoid ferrite, pro-eutectoid cementite, bainite and martensite in order to make the area rate of pearlite structure having a pearlite lamelar spacing of 200 nm or less to be 80 % or more and is made to have a tensile strength of 1,200 N/mm^2 or more and excellent delayed fracture resistance by using strong wire-drawing, the high strength wire material is cut into a predetermined length, a bolt head is formed on one end thereof by warm forging

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and the other end is thread-rolled or threaded.

[0008] Further, it is effective that the inventive high strength bolt contains (1) Si: 2.0 % or less (but not 0 %) and/or Co: 0.5 % or less (but no 0 %) or (2) Cr: 1.0 % or less (but not 0 %), according to the need.

[0009]

[Embodiments] The present inventors conducted investigations from various aspects in order to identify the cause of the delayed fracture of conventional steels for the production of a high strength bolt. As a result, it was found that there was a limitation to the improvement in the delayed fracture resistance in the conventional improvement method, which intended to avoid the temper brittleness zone, reduce the grain boundary segregation elements and reduce the crystal grain size by converting the structure to tempered martensite.

[0010] Hence, the present inventors conducted intensive research in order to further improve the delayed fracture resistance and, as a result, discovered that a high strength bolt having excellent delayed fracture resistance could be obtained by producing a bolt using a high strength wire material which consisted of a pearlite structure having a specific restriction and was made to have a strength of 1,200 N/mm^2 or more by strong wire-drawing. The present invention has thus been completed.

[0011] The high strength wire material for use in the present invention must restrain the generation of one or more structures selected from the group consisting of pro-eutectoid ferrite, pro-eutectoid cementite, bainite and martensite and must also be made to make the area rate of pearlite structure having a pearlite lamelar spacing of 200 nm or less to be 80 % or more.

Of the above described structures, the increase in the amounts of pro-eutectoid ferrite and pro-eutectoid cementite makes strong wire-drawing difficult because longitudinal crack may form during the wire drawing process, whereby it may become difficult to obtain a strength of 1,200 N/mm^2 or more by strong wire-drawing. Further, pro-eutectoid cementite and martensite, which may cause frequent disconnection during the wire drawing process, should be reduced in the amount. In addition, bainite, which is smaller in work-hardening amount than pearlite and ineffective in increasing the strength by strong wire-drawing, should be reduced in the amount.

[0012] In contrast to these structures, the amount of the pearlite structure, which is effective in trapping hydrogen at the interface between cementite and ferrite and in reducing the hydrogen accumulated in the grain boundary, should be increased as much as possible. Therefore, the area rate of the pearlite structure must be adjusted at 80 % or more by restraining the generation of one or more structures selected from the group consisting of pro-eutectoid ferrite, pro-eutectoid cementite, bainite and martensite as much as possible (in other words, less than 20 %). More specifically, the area rate of the pearlite structure must be adjusted at 80 % or more by reducing the amount of at least one structure selected from the group consisting of pro-eutectoid ferrite, pro-eutectoid cementite,

bainite and martensite as much as possible so that the total area rate of the structures becomes less than 20 %. The area rate of the pearlite structure is preferably 90 % or more and is more preferably 100 %.

[0013] The pearlite structure is required to have a pearlite lamelar spacing of 200 nm or less. Micronizing pearlite lamelar spacing is effective for strengthening the steel material and also increases the boundary between cementite and ferrite as described above, thereby promoting the effect on trapping hydrogen. In order to sufficiently obtain the above described effect, the pearlite lamelar spacing must be 200 nm or less. The pearlite lamelar spacing is preferably 150 nm or less, more preferably 100 nm or less and even more preferably 75 nm or less.

[0014] Since it is impossible to obtain the required dimensional accuracy by simply rolling or forging a wire rod which can be used in the present invention as a starting material and it is also impossible to ensure a strength of 1,200 N/mm^2 or more, conducting strong wire-drawing is required. Further, by conducting strong wire-drawing, cementite in a part of pearlite can be finely dispersed, the effect on trapping hydrogen can be improved and resists the progress of cracking by aligning the structures in the wire-drawing direction (the crack propagation direction is perpendicular to the wire-drawing direction).

[0015] The inventive high strength bolt is assumed to be produced using a medium carbon alloy steel containing 0.5 to 1.0 % C. The reason for limiting the range of C content is as follows.

[0016] <u>C: 0.5 to 1.0 %</u>

C is an economical element effective in increasing the strength of the steel, and an increase in C content leads to increase in strength. In order to achieve a desirable strength of the steel, the C content must be 0.5 % or more. However, if the C content exceeds 1.0 %, the amount of the pro-eutectoid cementite precipitated may increase, resulting in significant deterioration in toughness and ductility and also in wire-drawing processability. Thus, the lower limit of the C content is 0.65 % and is preferably 0.7 %. The upper limit of the C content is preferably 0.95 % and is more preferably 0.9 %.

[0017] The inventive high strength bolt can of course contain various elements which are commonly used (such as Si, Co, Mn, Cu, Ni, Cr, Mo, Ti, Nb, V, W, Al, and B); in particular, the addition of Si or Co in a specific amount is effective on restraining the precipitation of pro-eutectoid cementite, and the addition of Cr is effective on refining the pearlite lamelar spacing, thereby improving the strength of the wire material and improving wire-drawing processability. The reasons for limiting the elements which can be optionally added are as follows.

[0018] Si: 2.0 % or less (but not 0 %)

Si improves the hardenability of the steel wire, thereby restraining the precipitation of pro-eutectoid cementite. Further, Si is expected to produce an effect as a deoxidizer and is solid-solubilized in ferrite, thereby producing a marked action to strengthen the solid solution. The above described effects increase as the Si content increases, but an excessive content may lead to a deterioration of the ductility of the steel wire after the wire-drawing process; therefore, the upper is set to 2.0 %.

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The upper limit of the Si content is preferably 1.0 % and is more preferably 0.5 %.

[0019] Co: 0.5 % or less (but not 0 %)

Similar to Si, Co also has an effect on restraining the precipitation of pro-eutectoid cementite and is a particularly effective constituent in the inventive high strength bolt which intends to reduce the content of pro-eutectoid cementite. The above described effect increases as the content increases, but the effect may become saturated at a content of over 0.5 %, which is uneconomical; therefore, the upper limit is set to 0.5 %. The Co content is preferably in a range of from 0.05 to 0.3 %, and it is more preferred that the lower limit is set to 0.1 % and the upper limit is set to 0.2 %.

[0020] Cr: 1.0 % or less (but not 0 %)

Cr is effective on refining the pearlite lamelar spacing, thereby improving the strength of the wire material and improving wire-drawing processability. However, an excessive content of Cr leads to elongation of the period until completion of transformation and thus to expansion of facility and decrease in productivity; therefore, the upper limit is set to 1.0 %. The lower limit of the Cr content is preferably 0.05 % and is more preferably 0.1 %. The upper limit of the C content is preferably 0.5 % and is more preferably 0.3 %.

[0021] Mn: 0.2 to 1.0 %

Mn has an effect as a deoxidizer and an effect on enhancing the hardenability of the steel wire, thereby improving the uniformity of the structures in the steel wire. In order to obtain the above described effects, the content must be 0.2 % or more. However, an excessive Mn content leads to the generation of super-cooled structures such as martensite and bainite in the Mn segregation region and hence to deterioration in wire-drawing processability; therefore, the upper limit is set to 1.0 %. The lower limit of the Mn content is preferably 0.40 % and is more preferably 0.45 %. The upper limit of the Mn content is preferably 0.70 % and is more preferably 0.55 %.

[0022] Cu: 0.5 % or less (but not 0 %)

Cu is an element effective in improving the strength of the steel wire by its precipitation-hardening action. However, an excessive Cu content causes embrittlement of grain boundary and thus deterioration in delayed fracture resistance; therefore, the upper limit is set to 0.5 %. The lower limit of the Cu content is preferably 0.05 % and is more preferably 0.1 %. The upper limit of the Cu content is preferably 0.3 % and is more preferably 0.2 %.

[0023] <u>Ni: 1.0 % or less (but not 0 %)</u>

Ni is not effective in improving the strength of the steel wire, but is effective in increasing the toughness of drawn wire rod. However, as in the case with Cr, an excessive content of Ni leads to elongation of the period until completion of transformation and thus to expansion of facility and decrease in productivity; therefore, the upper limit is set to 1.0 %. The lower limit of the Ni content is preferably 0.05 % and is more preferably 0.1 %. The upper limit of the Ni content is preferably 0.5 % and is more preferably 0.3 %.

[0024] <u>One or more element selected from the group consisting of Mo, Ti, Nb, V, and W: total 0.01 to 0.5 %</u>

These elements form micro carbides/nitrides, which are effective in improving delayed fracture resistance. The carbides and nitrides of these elements are effective in reducing the size of crystal grain. In order to obtain the above described effects,

the total content of these elements must be 0.01 %, but an excessive total content leads to inhibition of the delayed fracture resistance and also to deterioration in toughness; therefore, the total content is set to 0.5 % or less. The lower limit of the total content of these elements is preferably 0.02 % and is more preferably 0.03 %. The upper limit of the total content of these elements is preferably 0.3 % and is more preferably 0.1 %.

[0025] Al: 0.01 to 0.05 %

Al is effective in improving the delayed fracture resistance by capturing N in steel to form AlN and reducing the size of crystal grain. In order to obtain the above described effects, the content must be 0.01 % or more, but if the content exceeds 0.05 %, nitride inclusions or oxide inclusions may be generated and wire-drawing efficiency may deteriorate; therefore, the content must be 0.05 % or less. The lower limit of the Al content is preferably 0.025 % and is more preferably 0.035 %.

[0026] B: 0.0005 to 0.003 %

B is added for improvement in the quenching efficiency, but the content must be 0.0005 % or more in order to obtain the effect. However, an excessive content, which is a content exceeding 0.003 %, leads to deterioration in toughness. The lower limit of the B content is preferably 0.0010 % and is more preferably 0.0025 %.

[0027] N: 0.015 % or less (but not 0 %)

N forms a nitride, such as AlN and TiN, thereby reducing the size of crystal grain and thus improving the delayed fracture resistance. However, an excessive content not only excessively increases the proportion of nitride, resulting in deterioration in wire-drawing efficiency but also may promote the ageing in the wire drawing process due to the solution N; therefore, the content must be 0.015 % or less. The upper limit of the N content is preferably 0.007 % and is more preferably 0.005 %.

[0028] In the inventive high strength bolt, the constituents other than the above described elements (in other words, balance) basically consists of iron, but small amounts of other elements can also be contained and such a case including other elements is also included in the scope of the present invention. From the viewpoint of further improving the characteristics of the present invention, the content of P, S and O can be restrained as described below. Further, the inventive high strength wire rod contains inevitable impurities, but the content of such inevitable impurities is acceptable without impairing the scope of the present invention.

[0029] P: 0.03 % or less (including 0 %)

P is an element which causes grain boundary segregation and thus deterioration in delayed fracture resistance. Hence, the delayed fracture resistance can be improved by limiting the P content to

0.03 % or less. The P content is preferably reduced to 0.015 % or less and is more preferably reduced to 0.005 % or less.

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[0030] S: 0.03 % or less (including 0 %)

S forms MnS in steel, and MnS becomes the stress-concentration point when a stress is applied. Hence, the S content is preferably as small as possible in order to improve the delayed fracture resistance and is therefore preferably limited to 0.03 % or less. The S content is preferably reduced to 0.01 % or less and is more preferably reduced to 0.005 % or less.

[0031] O: 0.005 % or less (including 0 %)

O is hardly soluble in steel at ambient temperature but is present in the form of a hard oxide inclusion, which may become a cause of cuppy breakage in the wire drawing process. Hence, the O content should be as small as possible and must be reduced at least to 0.005 % or less. The O content is preferably reduced to 0.003 % and is more preferably reduced to 0.002 % or less.

[0032] The structure of the high strength wire material used in the present invention can be modified by various methods, and typical methods are described in the following. An example of such typical methods is a method wherein a steel material having the above described chemical constituents is first subjected to a hot rolling or hot forging so that the final rolling or forging temperature becomes 800 degrees Celsius or more, the

resulting steel material is continuously cooled to a temperature of 400 degrees Celsius so that the average cooling rate V (°C/sec) satisfies the following inequality (1), and the resulting cooled steel material is subsequently allowed to stand to cool.

$$166 \times (\text{wire diameter})^{-1.4} \leqq V \leqq 288 \times (\text{wire diameter})^{-1.4} \ldots (1)$$

[0033] Through the above described processes, a pearlite structure which is more uniform than a conventional rolled material can be obtained and the strength before the wire-drawing process can be improved. If the final rolling or forging temperature is too low, austenitizing may become insufficient, resulting in a uniform pearlite structure not being obtained; therefore, the terminal temperature must be 800 degrees Celsius or more. The terminal temperature is preferably in a range of from 850 to 950 degrees Celsius, and is more preferably in a range of from 850 to 900 degrees Celsius.

[0034] If the average cooling rate V is smaller than $166 \times (\text{wire diameter})^{-1.4}$, not only may a uniform pearlite structure be obtained but also pro-eutectoid ferrite and pro-eutectoid cementite may be readily generated. If the average cooling rate V is greater than $288 \times (\text{wire diameter})^{-1.4}$, bainite and martensite may be readily generated.

[0035] Further, another example of the typical methods is a method wherein a steel material having the above described chemical constituents is first heated to a temperature of 800 degrees Celsius or more, is then cooled to a temperature in arrange of from 520 to 650 degrees Celsius, and is isothermally retained at the temperature (patenting treatment). Through the processes, a pearlite structure which is more uniform than a conventional rolled material can also be obtained and the strength before the wire-drawing process can be improved.

[0036] In the above described method, the heating temperature for the steel material must be specified to 800 degrees Celsius or more for the same reason as the above described final rolling or forging temperature. The preferred heating temperature range is the same as described above. The patenting treatment uses a salt bath, lead, a fluidized-bed and the like and it is preferred to rapidly cool the heated wire material at as fast a cooling rate as possible. In order to obtain a uniform pearlite structure, the wire material must be isothermally transformed at a temperature in a range of from 520 to 650 degrees Celsius. The preferred temperature range of the isothermal transformation is from 550 to 600 degrees Celsius and the isothermal retention temperature is most preferably neat the pearlite nose point in the TTT graph.

[0037] Further, another example of the typical methods is a method wherein a steel material is subjected to hot rolling or hot forging so that the final rolling or forging temperature becomes 800 degrees Celsius or more, the resulting steel material is cooled to a temperature in a range of from 520 to 750 degrees Celsius at a cooling rate of 5 °C/sec or more, the resulting steel material is then retained at an average cooling temperature of 1.0 °C/sec or less for 200 seconds or more, and the resulting cooled steel material is subsequently allowed to stand to cool. Through the processes, a pearlite structure which is more uniform than a conventional rolled material can also be obtained and the strength before the wire-drawing process can be improved. The operations of each step in the above described methods are as follows.

[0038] The temperature range after hot rolling or forging temperature is specified as 800 degrees Celsius or more for the same reason as the steel material heating temperature. The preferred final rolling or forging temperature range is the same as described above. If the cooling rate after hot rolling and forging is too slow, ferrite transformation may be caused during cooling; therefore, the cooling rate is preferably as fast as possible. Therefore, the cooling rate is specified at 5 °C/sec or more. The cooling rate is preferably 10 °C/sec or more and is more preferably 30 °C/sec or more. The steel material must be

cooled to a temperature in a range of from 520 to 750 degrees Celsius by cooling under the above described conditions; however, if the final cooling temperature is less than 520 degrees Celsius or exceeds 750 degrees Celsius, structures other than pearlite can be readily formed during the following slow cooling process.

[0039] After cooling as described above, from the viewpoint of obtaining a uniform pearlite structure, the steel material must be retained for 200 seconds or more while cooling the steel material at an average cooling rate of 1.0 °C/sec or less from the above-specified temperature (520 to 750 degrees Celsius: slow cooling initiation temperature). If the average cooling rate is faster than 1.0 °C/sec or the retention period is less than 200 seconds, the slow cooling may be conducted before pearlite transformation and bainite and martensite may be readily formed. Further, the cooling rate is preferably 0.5 °C/sec or less and is more preferably 0.2 °C/sec or less. Further, the retention period is preferably 300 seconds or more and is more preferably 600 seconds or more. The retention is most preferably conducted at a temperature near the pearlite nose point in the TTT graph for an extended period of time.

[0040] The high strength wire material thus obtained is first cut into a predetermined length, and (1) both ends are threaded by thread rolling or cutting (in other words, formed into a stud

bolt) or (2) one end is subjected to hot forging to form a bolt head and the other end is threaded by thread rolling or cutting before or after the hot forging, thereby obtaining a bolt having excellent delayed fracture resistance and high strength.

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In the method (2), the reason for adopting hot forging to form a bolt head is that it is difficult to form the wire material into the predetermined bolt shape by conventional cold forging due to the high strength of the wire material.

[0041] The present invention is described in greater detail with reference to embodiments. The embodiments do not restrict the present invention in any way and various modifications according to the scope of the present invention described above and below are also included in the present invention.

[0042]

[Detailed Description of Embodiments] Embodiment 1

Test steels respectively having the chemical compositions shown in the following Table 1 were hot-rolled into wires having a wire diameter of from 11 mmφ or 14 mmφ so that the final rolling temperature became approximately 930 degrees Celsius, and the resulting wires were cooled by blasting air at an average cooling rate of from 4.2 to 12.1 °C/sec (as shown in Table 2). Thereafter, the resulting wires respectively were drawn to a wire diameter of 7.06 mm (wire drawing rate: 59 % or 75 %).

[0043]

[Table 1]

Sample steels	Chemical constituents (% by mass)								
	C	Si	Mn	P	S	Al	N	O	Others
B	0.60	0.83	0.49	0.008	0.003	0.030	0.004	0.0007	-
C	0.82	0.89	0.50	0.005	0.002	0.031	0.004	0.0007	-
D	0.97	0.48	0.49	0.005	0.004	0.029	0.005	0.0008	-
E	1.30	1.90	0.53	0.005	0.003	0.031	0.005	0.0007	-
F	0.87	1.31	0.49	0.006	0.003	0.030	0.006	0.0007	-
G	0.90	2.23	0.50	0.006	0.003	0.033	0.006	0.0006	-
H	0.88	0.83	0.10	0.005	0.003	0.031	0.006	0.0055	-
I	0.87	0.85	1.22	0.006	0.002	0.030	0.006	0.0005	-
J	0.93	0.75	0.72	0.006	0.004	0.032	0.004	0.0008	Co: 0.41
K	0.85	0.97	0.52	0.006	0.002	0.030	0.005	0.0006	Cr: 0.32
L	0.86	0.96	0.53	0.006	0.002	0.030	0.005	0.0006	Cr: 1.22
M	0.34	0.19	0.70	0.006	0.004	0.033	0.003	0.0009	Cr: 0.95, Mo: 0.18

[0044] By using the resulting wires, M 8 × P 1.25 stud bolts as shown in Fig. 1 were produced and were then subjected to a delayed fracture resistance test. The delayed fracture test was conducted by first dipping the bolt in an acid (15 % HCl × 30 minutes), washing the resulting bolt with water, drying it, and leaving it in air under stress (loaded stress: 90 % of tensile strength), and the presence or absence of fracture was evaluated after 100 hours. In studying the structure, the pro-eutectoid ferrite, pro-eutectoid cementite, bainite and martensite or pearlite structure was separated by the following method; and the areal rate of each structure was determined. Further, the pearlite lamelar spacing was also measured by the following

method. For comparison, one sample steel wire was converted into the martensite structure by quenching and tempering and was then subjected to the delayed fracture resistance test (No. 13 in Table 2).

[0045] (Separation of each structure) The cross-sectional face of a steel wire was embedded, polished and corroded by dipping it in alcoholic 5 % picric acid solution for 15 to 30 seconds; and the structure of the D/4 region (D: diameter) was observed under a scanning electron microscope (SEM). Photographs of 5 to 10 visual fields were taken at a magnification of 1,000 to 3,000 times for determining the pearlite structure region, and the area rate of each structure was determined by using an image-analyzer. As for the bainite and pro-eutectoid ferrite structures, which are less easily differentiated from the pearlite structure, the structure shown in Fig. 2 (photo replacing drawing) was regarded as the bainite structure, and the structure shown in Fig. 3 (photo replacing drawing) as the pro-eutectoid ferrite structure. In general tendency of these structures, pro-eutectoid ferrite and pro-eutectoid cementite precipitated along the original austenite grain boundary while martensite precipitated in the bulky shape.

[0046] (Measurement method for pearlite lamelar spacing) The cross-sectional face of a steel wire was embedded, polished and corroded by dipping it in alcoholic 5 % picric acid solution for

15 to 30 seconds; and the structure of the D/4 region (D: diameter) was observed under a scanning electron microscope (SEM). Photographs of 10 visual fields of the portion where the lamelar spacing was considered to be narrowest in the pearlite structure neat the D/4 region were taken at a magnification of 5,000 to 100,000 times, and the length of a line which vertically crossed each lamelar was measured. The average value of 10 visual fields was specified as the average pearlite lamelar spacing.

[0047] The structures of each wire material are shown in the following Table 2 together with the average cooling rate V, and the results of the delayed fracture resistance test are shown in the following Table 3 together with wire-drawing conditions and mechanical characteristics. The appropriate range of the average cooling rate [in other words, the range which satisfies the above described inequality (1)] is $4.12 \leqq V \leqq 7.16$ (°C/sec) in the case of the wire diameter being 14 mm and is $5.78 \leqq V \leqq 10.03$ (°C/sec) in the case of the wire diameter being 11 mm.

[0048]

[Table 2]

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Test No.	Sample steels	Pro-eutectoid ferrite area rate (%)	Pro-eutectoid cementite area rate (%)	Bainite area rate (%)	Martensite area rate (%)	Pearlite fraction area rate (%)	Pearlite lamelar spacing (nm)	Average cooling rate V (°C/sec)	Note
1	C	27	0	0	0	73	152	4.2	Comparative Example
2	C	0	0	15	12	77	82	12.1	Comparative Example
3	B	18	0	0	0	82	118	6.2	Embodiment
4	C	9	0	0	0	91	92	8.7	Embodiment
5	D	31	11	0	0	86	78	8.5	Embodiment
6	E	0	35	0	0	65	67	8.6	Comparative Example
7	F	6	0	0	0	94	99	8.7	Embodiment
8	G	4	5	0	0	91	106	8.5	Reference Example
9	H	10	0	0	0	90	72	9.2	Reference Example
10	I	0	0	11	25	64	103	8.5	Comparative Example
11	J	3	9	0	0	88	65	8.3	Embodiment
12	K	0	0	0	0	100	82	8.7	Embodiment
13	L	0	0	9	23	68	73	8.6	Comparative Example
14	M	880 °C × 30 min. → OQ, 460 °C × 90 min. → WC (100 % tempered martensite)						-	Comparative Example

[0049]

[Table 3]

Test No.	Initial wire diameter (mm)	Initial strength (N/mm^2)	Final wire diameter (mm)	Final strength (N/mm^2)	Wire drawing rate (%)	Wire-drawing efficiency	Delayed fracture resistance	Note
1	11.0	954	7.06	1311	59	Good	×	Comparative Example
2	11.0	1221	7.06	1578	59	Good	×	Comparative Example
3	14.0	814	7.06	1217	75	Good	○	Embodiment
4	11.0	1139	7.06	1496	59	Good	○	Embodiment
5	11.0	1213	7.06	1634	59	Good	○	Embodiment
6	11.0	1714	7.06	Unable to be drawn due to breakage		Breakage	-	Comparative Example
7	11.0	1298	7.06	1677	59	Good	○	Embodiment
8	11.0	1562	7.06	Unable to be drawn due to breakage		Breakage	-	Reference Example
9	11.0	1097	7.06	Unable to be drawn due to breakage		Breakage	-	Reference Example
10	11.0	1365	7.06	Unable to be drawn due to breakage		Breakage	-	Comparative Example
11	11.0	1288	7.06	1693	59	Good	○	Embodiment
12	11.0	1204	7.06	1573	59	Good	○	Embodiment
13	11.0	1221	7.06	Unable to be drawn due to breakage		Breakage	○	Embodiment
14	11.0	-	7.06	1318	-	-	×	Comparative Example

[0050] Embodiment 2

The test steel C shown in Table 1 was hot-rolled into a wire having a wire diameter of 11 mmφ so that the final rolling temperature became approximately 930 degrees Celsius, the resulting wire was then rapidly cooled, and was subjected to a patenting treatment under the conditions shown in the following Table 4 (heating temperature: 750 to 935 degrees Celsius; isothermal transformation: 495 to 670 degrees Celsius × 4

minutes). Thereafter, the resulting wire was drawn to a wire diameter of 7.06 mm (wire drawing rate: 59 %).

[0051]

[Table 4]

Test No.	Heating temperature on patenting (°C)	Isotherm-al retention (°C)	Pro-eutectoid ferrite area rate (%)	Pro-eutectoid cementite area rate (%)	Bainite area rate (%)	Marten-site area rate (%)	Pearlite fraction area rate (%)	Pearlite lamelar spacing (nm)	Note
15	935	560	2	0	0	0	98	78	Embodiment
16	750	550	41	5	9	0	59	72	Comparative Example
17	930	670	25	0	0	0	75	223	Comparative Example
18	930	495	0	0	30	3	57	62	Comparative Example

[0052] By using the resulting wire materials, M 8 × P 1.25 stud bolts as shown in Fig. 1 were produced and were then subjected to the same delayed fracture resistance test as in the embodiment 1. The structures of each wire are shown in the Table 4, and the results of the delayed fracture resistance test are shown in the following Table 5 together with wire-drawing conditions and mechanical characteristics.

[0053]

[Table 5]

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Test No.	Initial wire diameter (mm)	Initial strength (N/mm^2)	Final wire diameter (mm)	Final strength (N/mm^2)	Wire drawing rate (%)	Wire-drawing efficiency	Delayed fracture resistance	Note

15	11.0	1227	7.06	1594	59	Good	○	Embodiment
16	11.0	1137	7.06	1494	59	Good	×	Comparative Example
17	14.0	1188	7.06	1545	59	Good	×	Comparative Example
18	11.0	1284	7.06	Unable to be drawn due to breakage		Breakage	-	Comparative Example

[0054] <u>Embodiment 3</u>

The test steel C shown in Table 1 was hot-rolled into a wire having a wire diameter of 11 mmφ under the rolling conditions shown in the following Table 6. Thereafter, the resulting wire was drawn to a wire diameter of 7.06 mm (wire drawing rate: 59 %).

[0055]

[Table 6]

Test No.	Final rolling temperature (°C)	Cooling rate after rolling (°C/sec)	Slow cooling initiation temperature (°C)	Cooling rate for slow cooling (°C/sec)	Retention period (sec)	Slow cooling initiation temperature (°C)	Note
19	930	30	570	0.2	250	520	Embodiment
20	930	25	680	0.8	250	480	Embodiment
21	930	30	560	0.1	800	485	Embodiment
22	750	25	570	0.2	250	520	Comparative Example
23	935	3	570	0.2	250	520	Comparative Example
24	930	15	800	0.2	250	750	Comparative Example
25	935	35	500	0.2	250	455	Comparative Example
26	930	25	630	0.1	250	330	Comparative Example
27	925	25	570	0.2	150	540	Comparative Example

[0056] By using the resulting wire, M 8 × P 1.25 stud bolts as shown in Fig. 1 were produced and were then subjected to the same delayed fracture resistance test as in the embodiment 1. The structures of each wire are shown in the following Table 7, and the results of the delayed fracture resistance test are shown in the following Table 8 together with wire-drawing conditions and mechanical characteristics.

[0057]

[Table 7]

Test No.	Pro-eutectoid ferrite area rate (%)	Pro-eutectoid cementite area rate (%)	Bainite area rate (%)	Martensite area rate (%)	Pearlite fraction area rate (%)	Pearlite lamelar spacing (nm)	Note
19	4	0	0	0	96	82	Embodiment
20	8	0	0	0	92	105	Embodiment
21	5	0	0	0	95	71	Embodiment
22	42	0	10	0	58	80	Comparative Example
23	44	0	0	0	56	82	Comparative Example
24	29	0	0	0	71	235	Comparative Example
25	0	0	32	10	58	68	Comparative Example
26	0	0	26	15	69	203	Comparative Example
27	0	0	10	39	51	81	Comparative Example

[0058]

[Table 8]

Test No.	Initial wire diameter (mm)	Initial strength (N/mm^2)	Final wire diameter (mm)	Final strength (N/mm^2)	Wire drawing rate (%)	Wire-drawing efficiency	Delayed fracture resistance	Note
19	11.0	1245	7.06	1602	59	Good	○	Embodiment
20	11.0	1249	7.06	1605	59	Good	○	Embodiment
21	11.0	1231	7.06	1588	59	Good	○	Embodiment
22	11.0	1132	7.06	1489	59	Good	×	Comparative Example
23	11.0	1164	7.06	1520	59	Good	×	Comparative Example
24	11.0	1188	7.06	Unable to be drawn due to breakage		Breakage	-	Comparative Example
25	11.0	1283	7.06	Unable to be drawn due to breakage		Breakage	-	Comparative Example
26	11.0	1312	7.06	Unable to be drawn due to breakage		Breakage	-	Comparative Example
27	11.0	1360	7.06	Unable to be drawn due to breakage		Breakage	-	Comparative Example

[0059] As is clear from the results shown above, the bolt which satisfies the requirement specified by the present invention is found to have excellent delayed fracture resistance while having a tensile strength of 1,200 N/mm^2 or more.

[0060]

[Effect of the Invention] In accordance with the structure of the present invention,

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a high strength bolt having excellent delayed fracture resistance while having a tensile strength of 1,200 N/mm^2 can be obtained.

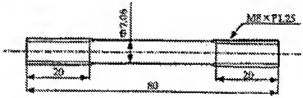
[Brief Description of the Drawings]

[Fig. 1] Schematic diagram illustrating the bolt shape which was subjected to the delayed fracture resistance test in the embodiments.

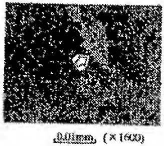
[Fig. 2] Photo replacing drawing illustrating bainite structure.

[Fig. 3] Photo replacing drawing illustrating pro-eutectoid ferrite structure.

[Fig. 1]



[Fig. 2]



[Fig. 3]